Standard Operating Procedures for Analysis of Total Chlorinated Ethenes in Soil and Groundwater Using the Color Tec Screening Method

Description

This SOP describes a field screening method for determining total chlorinated ethene concentrations in the headspace above soil and groundwater samples. The method employs colorimetric gas detector tubes to analyze the headspace gases. To conduct the method, groundwater or a soil and water mixture are placed in a standard 40 mL VOA vial and capped. The sample vial is then agitated vigorously for 20 seconds. Using a manual vacuum pump, two hollow needles, and Tygon® tubing, the headspace vapor is purged from the vial through the colorimetric gas detector tube. The tips of the needles are positioned so that one is in the headspace area and one is submerged at the bottom of the vial. As air is drawn from the headspace through one needle, ambient air is allowed into the bottom of the vial through the other needle. This process purges the VOCs from the water so no headspace calculation using Henry's Constant is required.

Each colorimetric tube contains a catalyst that decomposes the chlorinated ethene, releasing hydrogen chloride, which discolors the reagent (4-phenylazo-diphenylamine) in the tube. Any color change within the detector tube indicates the presence of a chlorinated ethene. The detector tubes are constructed of glass and printed with calibration scales to facilitate measurement of the linear extent of the reaction within the tube. The manufacturer of the tubes (Gastec®) provides tubes for a variety of concentration ranges. The lowest concentration range tube is used initially to screen the sample. When a positive result is observed, the concentration level is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube. If the calibrated range of the tube is exceeded by the reaction, a tube with a higher concentration range is used to screen a duplicate sample. This procedure is repeated until the approximate concentration is determined.

Safety Precautions

Safety glasses should be worn whenever handling potentially contaminated soil or water.

Analysis Procedure

Materials Needed

Gastec® hand pump or equivalent
Extra low, low, medium, and high range Gastec® colorimetric tubes
Plastic male luer lock threaded fitting with ⅛-in hose barb
Plastic male luer non-threaded fitting with ⅛-in hose barb
Tygon® lab tubing ⅛-in ID × ¼-inch OD
Deflected noncoring septum penetration needle (18G × 4)
Hot plate
Test tube rack
Vortex mixer
Charcoal prefilter tubes
40 mL clear VOA with Teflon-silicon septa
Kimwipes™

Reagents Needed

For soil samples, 20 mL ultra-pure deionized water per sample
Chlorinated ethene QC standard
Decontamination materials

Pitfalls

- The color change indicated by a sample containing less than 10 µg/L PCE or less than 30 µg/L vinyl chloride is very subtle and could be missed by an inexperienced operator. The solution to this problem is practice and careful examination of each tube after pumping.

- Introduction of water vapor into the tube past the catalyst stage could indicate a low level false positive to an inexperienced operator. However, water-induced false positives are easily recognized by an experienced operator and are avoidable through proper placement of the extraction needle in the VOA vial.

- If chlorinated tap water is used in the soil sample screening procedure, the free chlorine in the tap water could (theoretically) indicate a low-level false positive in the soil sample. The potential for producing a false reading in this manner can be avoided by using ultrapure deionized water during the soil sample screening procedure.

- Do not use hydrochloric acid as a preservative when using this method.

Procedure

Water

1. Fill 40 mL VOA vial with 24 ml (about 60 percent of volume) of groundwater sample and cap.

2. Shake the sample vigorously for 20 seconds.

3. Penetrate the septum with a needle equipped with the charcoal pre-filter tube and position the needle point near the bottom of the vial.

4. Penetrate the septum with the extraction needle attached to the Tygon® tubing. Ensure that the needle tip is well clear of the water surface.

5. Break one end of the detection tube and attach it to the Tygon® tubing.

6. Break the other end of the detection tube and attach it to the hand pump.

7. Begin drawing air from the vial and observe any change in color in the tube.

8. Record the concentration reached by the change in color on the scale marked on the tube.

Soil

1. Fill 40-ml VOA vial with approximately 10 cc of soil and 10 ml of ultrapure deionized water (about 50-70 percent of volume) and cap.

2. Shake the sample vigorously for 20 seconds.

3. Penetrate the septum with a needle equipped with a charcoal pre-filter tube and position the needle point near the top of the soil.

4. Penetrate the septum with a needle attached to the Tygon® tubing. Ensure that the needle tip is well clear of the water surface.

5. Break one end of the detection tube and attach it to the Tygon® tubing.

6. Break the other end of the detection tube and attach it to the hand pump.

7. Begin drawing air from the vial and observe any change in color in the tube.

8. Record the concentration reached by the change in color on the scale marked on the tube.

Detection Limits

The practical quantitation limit for PCE is 5-10 ppb as measured in the headspace. This method does not employ Henry's Constant or other partitioning methods to back calculate the actual concentrations in the soil or water sample. Samples containing only trans-1,2-dichloroethene, 1,1-dichloroethene, or vinyl chloride are generally not detectable with ColorTec at concentrations below 25 µg/L.

General Interferences

Other compounds including bromine, free chlorine, and hydrogen chloride can also indicate a positive reaction within the detector tube.

The Gastec® tubes are sold to detect specific chlorinated alkenes. However, if there are other chlorinated ethenes present in a sample, the identification of a specific chlorinated compound is not possible using the ColorTec method. The developers of the ColorTec method recommend using the Gastec® tetrachloro-ethene tubes for all sampling.

The detector tubes are designed to be used at temperatures of 0 to 40 °C (32 to 104 °F) and are calibrated based on a tube temperature (not sample temperature) of 20 °C (68 °F). There is a significant reduction in sensitivity of the tubes when sustained ambient air temperatures are significantly lower than 20 °C while conducting the method. The reduction in the temperature of the reagent decreases the reaction time resulting in a less visible color change within the tube. Conversely, when ambient temperatures are in excess of 20 °C, the tubes may be hypersensitive. The following correction factors provided by Gastec® can be applied to the readings to correct for sustained ambient temperatures other than 20 °C or 68 °F:

Temperature	0 °C (32 °F)	10 °C (50 °F)	20 °C (68 °F)	30 °C (86 °F)	40 °C (104 °F)
Correction Factor	× 2	× 1.3	× 1	× 0.7	× 0.55

Because tube/sample temperatures below 20 °C significantly reduce the sensitivity of the method, it is recomended that the prepared sample vials and colorimetric tubes (prior to breaking the tips) be heated using a hot water bath prior to screening when ambient temperatures are below 20 °C.